JC17 Rec'd PCT/PTO 08 JUN 2001

FORM PTO-1390 (REV. 5-93)	U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER **10537/119**
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/857763

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATES CLAIMED
PCT/DE99/03942	**(09.12.99)** **09 December 1999**	**(10.12.98)** **10 December 1998**

TITLE OF INVENTION
PROCESS FOR COATING HOLLOW BODIES

APPLICANT(S) FOR DO/EO/US
PILLHÖFER, Horst; FRITSCH, Andreas; DAUTL, Thomas and SCHESNY, Guido

Applicants herewith submit to the United States Designated/Elected Office (DO/EO/US) the following items and other information

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) immediately rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (Unexecuted)
10. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
    ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☒ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information: An English translation of the International Search Report; Marked-up version of the Substitute Specification and first page of the published International Application WO 00/34547.

EXPRESS MAIL NO. : EL327550945US

U.S. APPLICATION NO. (if known, see 37 C.F.R.1.5) 09/857763	INTERNATIONAL APPLICATION NO. PCT/DE99/03942	ATTORNEY'S DOCKET NUMBER 10537/119

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17. ☒ The following fees are submitted:

Basic National Fee (37 CFR 1.492(a)(1)-(5)):
Search Report has been prepared by the EPO or JPO $860.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) ... $690.00

No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) $710.00

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO $1,000.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) $100.00

				CALCULATIONS	PTO USE ONLY
ENTER APPROPRIATE BASIC FEE AMOUNT =				$ 860.00	
Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				$	
Claims	Number Filed	Number Extra	Rate		
Total Claims	7 - 20 =	0	X $18.00	$	
Independent Claims	1 - 3 =	0	X $80.00	$	
Multiple dependent claim(s) (if applicable)			+ $270.00		
TOTAL OF ABOVE CALCULATIONS =				$ 860.00	
Reduction by ½ for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				$	
SUBTOTAL =				$ 860.00	
Processing fee of $130.00 for furnishing the English translation later the ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)). +				$	
TOTAL NATIONAL FEE =				$ 860.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property +				$	
TOTAL FEES ENCLOSED =				$ 860.00	
				Amount to be: refunded	$
				charged	$ 860.00

a. ☐ A check in the amount of $__________ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 11-0600 in the amount of **$860.00** to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 11-0600. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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26646
PATENT TRADEMARK OFFICE

SIGNATURE

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NAME

6/8/01
DATE

[10537/119]

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s)	:	Horst PILLHOEFER et al.
Serial No.	:	To Be Assigned
Filed	:	Herewith
For	:	PROCESS FOR COATING HOLLOW BODIES
Examiner	:	To Be Assigned
Art Unit	:	To Be Assigned

Assistant Commissioner for Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT AND
37 C.F.R. § 1.125 SUBSTITUTE SPECIFICATION STATEMENT

S I R:

Kindly amend the above-captioned application before examination, as set forth below.

IN THE SPECIFICATION AND ABSTRACT:

In accordance with 37 C.F.R. § 1.121(b)(3), a Substitute Specification (including the Abstract, but without claims) accompanies this response. It is respectfully requested that the Substitute Specification (including Abstract) be entered to replace the Specification of record.

IN THE CLAIMS:

On the first page of the claims, first line, change "Patent claims" to --WHAT IS CLAIMED IS:--.

Please cancel, without prejudice, claims 1 to 11 in the underlying PCT application. Please also cancel, without prejudice, claims 1 to 7 in the annex to the International Preliminary Examination Report.

Please add the following new claims:

--8. (New) A method for coating a hollow body, comprising the steps of:

contacting a powder mixture with an inner surface of the hollow body to be coated, the powder mixture including a metal donor powder, an inert filler powder and an activator powder, the activator powder including a metal halide; and

heating the powder mixture;

wherein a mean particle size of the inert filler powder is approximately equal to a mean particle size of the metal donor powder;

wherein the mean particle size of the metal donor powder and the mean particle size of the inert filler powder are greater than 40 μm; and

wherein a metal donor powder content is 10% to 25% by weight of the powder mixture.

9. (New) The method according to claim 8, wherein the metal donor powder includes an alloy having a donor metal content of 20% to 80% by weight.

10. (New) The method according to claim 8, wherein the metal donor powder includes a mixture of a first alloy having a donor metal content of 40% to 70% by weight and a second alloy having a donor metal content of 30% to 50% by weight.

11. (New) The method according to claim 8, wherein the powder mixture includes an activator powder content of 2% to 5% by weight.

12. (New) The method according to claim 8, wherein the metal halide of the activator powder includes a metal halide of a donor metal.

13. (New) The method according to claim 8, wherein the donor metal powder includes AlCr.

14. (New) The method according to claim 8, wherein the mean particle size of the metal donor powder and the mean particle size of the inert filler powder are approximately 150 μm.--.

REMARKS

This Preliminary Amendment cancels, without prejudice, claims 1 to 11 in the underlying PCT Application No. PCT/DE99/03942. This Preliminary Amendment further cancels, without prejudice, claims 1 to 7 in the annex to the International Preliminary Examination Report and adds new claims 8 to 14. The new claims, inter alia, conform the claims to U.S. Patent and Trademark Office rules and do not add any new matter to the application.

In accordance with 37 C.F.R. § 1.121(b)(3), the Substitute Specification (including the Abstract, but without the claims) contains no new matter. The amendments reflected in the Substitute Specification (including Abstract) are to conform the Specification and Abstract to U.S. Patent and Trademark Office rules or to correct informalities. As required by 37 C.F.R. §§ 1.121(b)(3)(iii) and 1.125(b)(2), a Marked Up Version of the Substitute Specification comparing the Specification of record and the Substitute Specification also accompanies this Preliminary Amendment. Approval and entry of the Substitute Specification (including Abstract) is respectfully requested.

The underlying PCT Application No. PCT/DE99/03942 includes an International Search Report, dated May 26, 2000, a copy of which is included. The Search Report includes a list of documents that were considered by the Examiner in the underlying PCT application.

The underlying PCT Application No. PCT/DE99/03942 also includes an International Preliminary Examination Report dated April 10, 2001, an English translation of the annexed pages thereto are enclosed herewith.

It is respectfully submitted that the subject matter of the present application is new, non-obvious and useful. Prompt consideration and allowance of the application are respectfully requested.

Respectfully submitted,

KENYON & KENYON

Dated: 9/8/01

By: [signature]

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[10537/119]

PROCESS FOR COATING HOLLOW BODIES

FIELD OF THE INVENTION

The **present** invention relates to a process for coating hollow bodies, in which a powder mixture comprising a metal donor powder, an inert filler powder and an activator powder is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the body, e.g.**,** [comprising] **including** an Ni-, Co- or Fe-base alloy, and is heated.

BACKGROUND INFORMATION

[The known] **Conventional** processes for the diffusion coating of components made from heat-resistant alloys, such as Ni-, Co- or Fe-base alloys, include [what are known as the] **so-called** powder pack processes. A process of this type is [disclosed] **described, for example** in [US] **U.S. Patent No.** 3,667,985, in which the component surfaces to be coated are brought into contact with a donor powder [comprising] **including** titanium and aluminum, to which an inert filler material and a halide activator are admixed, and is heated. [US] **U.S. Patent No.** 3,958,047 [has disclosed] **describes** a powder pack process in which the metallic component is brought into contact with a donor powder which contains aluminum and chromium and is diffusion-coated by heating.

These processes are particularly suitable for coating the outer surfaces of metallic components, producing layer thicknesses of between 50 and 100 μm. However, [the] **these** processes have inherent drawbacks when coating internal surfaces, and consequently the internal layer thicknesses which are achievable with relatively complicated geometrical forms with narrow gaps, tight angles or undercuts are limited and inadequate, generally being below 30 μm. A problem in this

respect is that the donor powders have only a low ability to flow and therefore do not sufficiently fill the cavities. Moreover, after the coating, the donor powder can only be removed from the cavities with difficulty, and it is not possible to avoid leaving residues, and also the donor powder sinters to the surfaces.

The abovementioned drawbacks of the powder pack processes can in part be eliminated using [what are known as] **so-called** gas diffusion coating processes. One process of this type is [known from US] **described in U.S. Patent No.** 4,148,275, in which a powder mixture which contains, for example, aluminum is arranged in a first chamber and the metallic components to be coated are arranged in a second chamber of a vessel. The coating gas is generated by heating the powder and, using a carrier gas, is deposited on the outer and inner surfaces of the components to be coated. However, [the] **these** gas diffusion coating processes have the drawback that the devices for carrying out the process, such as, for example, for the forced guidance of the coating gases, are complex and expensive compared to those used for the powder pack processes. Furthermore, [in this case too,] the internal layer thicknesses which can be achieved are limited, since the coating gas or the donor metal gas is depleted on its route through the cavities of the component and a layer thickness gradient is produced along the length of the cavity. Since process conditions mean that the layer thickness of the outer coating is greater than that of the inner coating, the service life of the component is limited on account of the thinner internal coating.

[US] **U.S. Patent No.** 4,208,453 [has disclosed] **describes** a process for the diffusion coating of the inner and outer surfaces of components, such as gas turbine blades, in which a powder mixture comprises 10% of chromium donor powder with a particle size of from 10 to 20 μm and 90% of alumina granules

with a particle size of from 100 to 300 μm. In addition, a metal halide is added as activator. This [disclosure] **process** does not deal with measures for increasing the layer thickness in cavities of complicated geometry.

[DE] **German Published Patent Application No.** 30 33 074 [A1 discloses] **describes** a process for the diffusion coating of the inner surface of cavities, in which a metallic workpiece can be coated with an aluminizing diffusion powder mixture comprising 15% of aluminum powder with a particle size of 40 μm and 85% of alumina powder with a particle size of approximately 200 to 300 μm, as well as an NH_4Cl powder.

[US] **U.S. Patent No.** 5,208,071 [discloses] **describes** a process for aluminizing a ferritic component with an alumina slurry, followed by heat treatment, the slurry [comprising] **including** at least 10% by weight of chromium, at least 10% by weight of inert filler material, at least 12% by weight of water, a binder and a halogen activator, and finally the coated ferritic component is heat-treated. The process technology involved means that the use of a slurry differs significantly from a powder pack coating process.

[GB] **British Published Patent Application No.** 2 109 822 [A has disclosed] **describes** a metal diffusion process with which diffusion coatings can be produced more quickly than in the powder pack process, the coating powder being in loose form and being kept in contact with the component to be coated, in particular including with its internal surface, by mechanical means during the heating. The composition of the coating powder may comprise 10 to 60% of chromium powder, 0.1 to 20% of chromium halide and alumina.

[The problem on which] **It is an object of** the present invention [is based is that of improving a] **to provide an**

improved powder pack process [of the generic type described in the introduction in such a way] **so** that the layer thicknesses of the internal coating are sufficiently great even in the case of cavities with relatively complex geometries.

SUMMARY

According to **one example embodiment of** the **present** invention, [the solution to this problem is characterized in that] the inert filler powder [is provided with] **has** a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

[The] **One** advantage **of the method according to the present invention** is that, when the particle sizes are selected in this way, it is possible to increase the specific density without agglomeration of the powder mixture, for example, [on account of] **due to** an excessively high metal donor powder content. It is also ensured that there is no premature depletion of the donor metal. A powder mixture of this type has [good] **sufficient** flow properties and, in tight corners, gains access to internal cavities which are to be coated. It is possible to coat hollow bodies, such as guide vanes and rotor blades of gas turbines made from heat-resistant Ni-, Co- or Fe-base alloys. Even in tight corners or recessed regions of the cavities, the layer thicknesses of the internal coating [lie] **are** in the range [from] **of** 50 to 110 μm, therefore ensuring that the internal coating functions as an oxidation-resistant and corrosion-resistant layer.

[In a preferred configuration, the] **The** metal donor powder and the inert filler powder [are provided with] **may have** a mean particle size of greater than 40 μm, so that it is possible to achieve [good] **sufficient** permeation of the coating gas through the bed of the powder mixture.

The powder mixture [is preferably provided with] **may include** a metal donor powder content of 10 to 25% by weight, in order to prevent agglomeration of the powder mixture and to ensure [good] **sufficient** permeation through the bed.

Furthermore, [it is expedient for] an alloy [with] **having** a donor metal content of 20 to 80% by weight [to] **may** be provided as the metal donor powder, so that a sufficiently [great] **large** layer thickness is ensured [on account of] **due to** the high donor metal content.

[It may be advantageous for a] **A** mixture of an alloy [with] **having** a donor metal content of 40 to 70% by weight and an alloy [with] **having** a donor metal content of 30 to 50% by weight [to] **may** be provided as the metal donor powder, so that the depletion of the metal donor in the two alloys takes place in steps, i.e., with a time delay.

The metal donor powder and the inert filler powder may be provided with a mean or average particle size of 150 μm. A powder mixture of this type has [good] **sufficient** flow properties and fills the cavities having the internal surfaces to be coated [well on account of] **due to** an advantageous specific bulk density. In addition, there is [good] **sufficient** permeation of the coating gas through the bed of the powder mixture.

[Further configurations of the invention are described in the subclaims.]

DETAILED DESCRIPTION

The **present** invention is explained in more detail below with reference to **specific** examples.

In a first example, the hollow body is a hollow turbine guide vane of a gas turbine, which is provided with an oxidation-resistant and corrosion-resistant layer.

The cavity has a length of approximately 160 mm. Its inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite end sections. To coat the inner surfaces of the guide vanes, a powder mixture [comprising] **including** approximately 20% by weight of metal donor powder and approximately 80% by weight of inert filler powder is provided. AlCr is selected as the metal donor powder, and Al_2O_3 is selected as the inert filler powder. The melting point of AlCr is at least approximately 100°C higher than the coating temperature of approximately 800°C - 1200°C, so that there is no diffusion bonding of the metal particles to one another or agglomeration.

An activator powder forms approximately 3% by weight, the powder selected being AlF_3, i.e., a halide compound. Another example of a suitable activator powder is $CrCl_3$. A compound of this type [has to] **should** have a low [vapour] **vapor** pressure at the coating temperature, so that it is retained throughout the entire coating process. Moreover, a halide compound of the donor metal, in this case aluminum, is used, in order to avoid agglomeration as a result of a chemical reaction of the halogen with the donor metal.

[The mean particle size of the inert filler powder is 100 μm, and is significantly greater than the particle size of the metal donor powder, which is 60 μm.] The aluminum content, i.e., the metal donor content, in the metal donor powder is 50% by weight.

The powder mixture which has been prepared in this [way] **manner** is introduced into the cavity of the guide vanes for the purpose of coating the internal surfaces. The subsequent

coating [takes place] **is performed** at 1080°C with a holding time of 6 h, while the external coating, i.e.**,** the coating of the outer surfaces of the guide vane, [can take place] **may be performed** simultaneously in a single-stage process using a conventional powder pack process or alternatively by [means of] a gas diffusion coating process.

In the internal coating which is deposited in this [way] **manner**, the Al content in the layer is between 30 and 35% by weight.

In a second example, an inert filler powder (Al_2O_3) [with] **having** a mean particle size of approximately 100 μm is [once] again selected, forming approximately 80% by weight of the powder mixture. As activator powder, AlF_3 forming approximately 3% by weight of a powder mixture is selected and admixed.

Unlike in [Example 1] **the first example**, the metal donor powder, which forms approximately 20% by weight of the powder mixture, [comprises] **includes** two fractions. The first fraction is an alloy [comprising] **including** AlCr, in which the aluminum content is 50% by weight. In the second fraction, the donor metal content, i.e.**,** the aluminum content, is lower, being 30% by weight. This measure [can] **may** be used to optimize the coating process [in such a manner] **so** that [first of all] the fraction with the lower Al content is depleted, but the coating process is continued by the fraction with the higher Al content. In this [way] **manner**, it is possible to increase the ductility of the layers on the inner surfaces of the guide vane.

The Al content in the inner layers is 24 to 28% by weight. The inner layer thicknesses are between 65 and 105 μm and are therefore significantly above the layer thicknesses which can be achieved with the conventional (powder pack) processes.

In a third example, the hollow body is a hollow turbine guide vane of a gas turbine, which is provided with an oxidation-resistant and corrosion-resistant layer by [means of] a powder pack coating process. The elongate cavity is approximately 180 mm long. The inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite, longitudinal-side end sections. To coat the inner surface of the guide vane, a powder mixture [comprising] **including** approximately 15% by weight of metal donor powder and just below 85% by weight of inert filler powder is provided. Depending on the particular application, the metal donor powder content may [lie] **be** in the range [from] **of** 10 to 25% by weight. The metal donor powder is AlCr, and the inert filler powder is Al_2O_3. The activator powder used is a halogen compound, such as AlF_3, forming approximately 3% by weight. The activator powder is therefore a halide compound of the donor metal Al.

The mean particle size of the inert filler powder is approximately equal to the mean particle size of the metal donor powder, being 150 μm. The proportion of the donor metal Al in the metal donor powder, which is an alloy, is 50% by weight. The specific density of a powder pack mixture is high not because of a high metal donor powder content, but rather because of the selected particle size distribution. With this bed of the powder pack mixture, there is sufficient permeation by the coating gases emanating from the halide compound.

To coat the inner surface of the turbine guide vane, the powder mixture which has been prepared in this [way] **manner** is introduced into its cavity. At the selected particle size distribution of the inert filler powder and of the metal donor powder, the bed has [good] **sufficient** flow properties and gains access even to the tight corners of the cavity. The subsequent coating [takes place] **is performed** at 1080°C for a holding time of 6 h. It may be [carried out] **performed** at the

same time as the external coating, i.e.**,** the coating of the outer surface of the turbine guide vane, which may [take place] **be performed** using a conventional powder pack process or also using a gas diffusion coating process. Generally, the coating is [carried out] **performed** on a plurality of turbine guide vanes simultaneously.

The Al content in the internal coating which has been deposited in this [way] **manner** is between 30 and 35% by weight and consequently in a highly advantageous range, i.e.**,** there is, for example, no embrittlement of the layer.

Even in tight corners or recessed regions of the cavities, the layer thicknesses are in the range [from] **of** 60 to 110 μm, thus ensuring that the internal coating function has protection against oxidation and corrosion.

[Abstract] **ABSTRACT**

A process for coating hollow bodies, in which a powder mixture [comprising] **including** a metal donor powder, an inert filler powder and an activator powder [comprising] **including** a metal halide is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the hollow body and is heated, in which process, in order to increase the internal layer thicknesses, the inert filler powder is provided with a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

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PROCESS FOR COATING HOLLOW BODIES

FIELD OF THE INVENTION

The present invention relates to a process for coating hollow bodies, in which a powder mixture comprising a metal donor powder, an inert filler powder and an activator powder is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the body, e.g., including an Ni-, Co- or Fe-base alloy, and is heated.

BACKGROUND INFORMATION

Conventional processes for the diffusion coating of components made from heat-resistant alloys, such as Ni-, Co- or Fe-base alloys, include so-called powder pack processes. A process of this type is described, for example in U.S. Patent No. 3,667,985, in which the component surfaces to be coated are brought into contact with a donor powder including titanium and aluminum, to which an inert filler material and a halide activator are admixed, and is heated. U.S. Patent No. 3,958,047 describes a powder pack process in which the metallic component is brought into contact with a donor powder which contains aluminum and chromium and is diffusion-coated by heating.

These processes are particularly suitable for coating the outer surfaces of metallic components, producing layer thicknesses of between 50 and 100 μm. However, these processes have inherent drawbacks when coating internal surfaces, and consequently the internal layer thicknesses which are achievable with relatively complicated geometrical forms with narrow gaps, tight angles or undercuts are limited and inadequate, generally being below 30 μm. A problem in this respect is that the donor powders have only a low ability to flow and therefore do not sufficiently fill the cavities. Moreover, after the coating, the donor powder can only be removed from the cavities with difficulty, and it is not

possible to avoid leaving residues, and also the donor powder sinters to the surfaces.

The abovementioned drawbacks of the powder pack processes can in part be eliminated using so-called gas diffusion coating processes. One process of this type is described in U.S. Patent No. 4,148,275, in which a powder mixture which contains, for example, aluminum is arranged in a first chamber and the metallic components to be coated are arranged in a second chamber of a vessel. The coating gas is generated by heating the powder and, using a carrier gas, is deposited on the outer and inner surfaces of the components to be coated. However, these gas diffusion coating processes have the drawback that the devices for carrying out the process, such as, for example, for the forced guidance of the coating gases, are complex and expensive compared to those used for the powder pack processes. Furthermore, the internal layer thicknesses which can be achieved are limited, since the coating gas or the donor metal gas is depleted on its route through the cavities of the component and a layer thickness gradient is produced along the length of the cavity. Since process conditions mean that the layer thickness of the outer coating is greater than that of the inner coating, the service life of the component is limited on account of the thinner internal coating.

U.S. Patent No. 4,208,453 describes a process for the diffusion coating of the inner and outer surfaces of components, such as gas turbine blades, in which a powder mixture comprises 10% of chromium donor powder with a particle size of from 10 to 20 μm and 90% of alumina granules with a particle size of from 100 to 300 μm. In addition, a metal halide is added as activator. This process does not deal with measures for increasing the layer thickness in cavities of complicated geometry.

German Published Patent Application No. 30 33 074 describes a process for the diffusion coating of the inner surface of cavities, in which a metallic workpiece can be coated with an aluminizing diffusion powder mixture comprising 15% of aluminum powder with a particle size of 40 μm and 85% of alumina powder with a particle size of approximately 200 to 300 μm, as well as an NH_4Cl powder.

U.S. Patent No. 5,208,071 describes a process for aluminizing a ferritic component with an alumina slurry, followed by heat treatment, the slurry including at least 10% by weight of chromium, at least 10% by weight of inert filler material, at least 12% by weight of water, a binder and a halogen activator, and finally the coated ferritic component is heat-treated. The process technology involved means that the use of a slurry differs significantly from a powder pack coating process.

British Published Patent Application No. 2 109 822 describes a metal diffusion process with which diffusion coatings can be produced more quickly than in the powder pack process, the coating powder being in loose form and being kept in contact with the component to be coated, in particular including with its internal surface, by mechanical means during the heating. The composition of the coating powder may comprise 10 to 60% of chromium powder, 0.1 to 20% of chromium halide and alumina.

It is an object of the present invention to provide an improved powder pack process so that the layer thicknesses of the internal coating are sufficiently great even in the case of cavities with relatively complex geometries.

<u>SUMMARY</u>

According to one example embodiment of the present invention, the inert filler powder has a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

One advantage of the method according to the present invention is that, when the particle sizes are selected in this way, it is possible to increase the specific density without agglomeration of the powder mixture, for example, due to an excessively high metal donor powder content. It is also ensured that there is no premature depletion of the donor metal. A powder mixture of this type has sufficient flow properties and, in tight corners, gains access to internal cavities which are to be coated. It is possible to coat hollow bodies, such as guide vanes and rotor blades of gas turbines made from heat-resistant Ni-, Co- or Fe-base alloys. Even in tight corners or recessed regions of the cavities, the layer thicknesses of the internal coating are in the range of 50 to 110 μm, therefore ensuring that the internal coating functions as an oxidation-resistant and corrosion-resistant layer.

The metal donor powder and the inert filler powder may have a mean particle size of greater than 40 μm, so that it is possible to achieve sufficient permeation of the coating gas through the bed of the powder mixture.

The powder mixture may include a metal donor powder content of 10 to 25% by weight, in order to prevent agglomeration of the powder mixture and to ensure sufficient permeation through the bed.

Furthermore, an alloy having a donor metal content of 20 to 80% by weight may be provided as the metal donor powder, so that a sufficiently large layer thickness is ensured due to the high donor metal content.

A mixture of an alloy having a donor metal content of 40 to 70% by weight and an alloy having a donor metal content of 30 to 50% by weight may be provided as the metal donor powder, so that the depletion of the metal donor in the two alloys takes place in steps, i.e., with a time delay.

The metal donor powder and the inert filler powder may be provided with a mean or average particle size of 150 μm. A powder mixture of this type has sufficient flow properties and fills the cavities having the internal surfaces to be coated due to an advantageous specific bulk density. In addition, there is sufficient permeation of the coating gas through the bed of the powder mixture.

DETAILED DESCRIPTION

The present invention is explained in more detail below with reference to specific examples.

In a first example, the hollow body is a hollow turbine guide vane of a gas turbine, which is provided with an oxidation-resistant and corrosion-resistant layer.

The cavity has a length of approximately 160 mm. Its inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite end sections. To coat the inner surfaces of the guide vanes, a powder mixture including approximately 20% by weight of metal donor powder and approximately 80% by weight of inert filler powder is provided. AlCr is selected as the metal donor powder, and Al_2O_3 is selected as the inert filler powder. The melting point of AlCr is at least approximately 100°C higher than the coating temperature of approximately 800°C - 1200°C, so that there is no diffusion bonding of the metal particles to one another or agglomeration.

An activator powder forms approximately 3% by weight, the powder selected being AlF_3, i.e., a halide compound. Another example of a suitable activator powder is $CrCl_3$. A compound of this type should have a low vapor pressure at the coating temperature, so that it is retained throughout the entire coating process. Moreover, a halide compound of the donor metal, in this case aluminum, is used, in order to avoid

agglomeration as a result of a chemical reaction of the halogen with the donor metal.

The aluminum content, i.e., the metal donor content, in the metal donor powder is 50% by weight.

The powder mixture which has been prepared in this manner is introduced into the cavity of the guide vanes for the purpose of coating the internal surfaces. The subsequent coating is performed at 1080°C with a holding time of 6 h, while the external coating, i.e., the coating of the outer surfaces of the guide vane, may be performed simultaneously in a single-stage process using a conventional powder pack process or alternatively by a gas diffusion coating process.

In the internal coating which is deposited in this manner, the Al content in the layer is between 30 and 35% by weight.

In a second example, an inert filler powder (Al_2O_3) having a mean particle size of approximately 100 μm is again selected, forming approximately 80% by weight of the powder mixture. As activator powder, AlF_3 forming approximately 3% by weight of a powder mixture is selected and admixed.

Unlike in the first example, the metal donor powder, which forms approximately 20% by weight of the powder mixture, includes two fractions. The first fraction is an alloy including AlCr, in which the aluminum content is 50% by weight. In the second fraction, the donor metal content, i.e., the aluminum content, is lower, being 30% by weight. This measure may be used to optimize the coating process so that the fraction with the lower Al content is depleted, but the coating process is continued by the fraction with the higher Al content. In this manner, it is possible to increase the ductility of the layers on the inner surfaces of the guide vane.

The Al content in the inner layers is 24 to 28% by weight. The inner layer thicknesses are between 65 and 105 μm and are therefore significantly above the layer thicknesses which can be achieved with the conventional (powder pack) processes.

In a third example, the hollow body is a hollow turbine guide vane of a gas turbine, which is provided with an oxidation-resistant and corrosion-resistant layer by a powder pack coating process. The elongate cavity is approximately 180 mm long. The inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite, longitudinal-side end sections. To coat the inner surface of the guide vane, a powder mixture including approximately 15% by weight of metal donor powder and just below 85% by weight of inert filler powder is provided. Depending on the particular application, the metal donor powder content may be in the range of 10 to 25% by weight. The metal donor powder is AlCr, and the inert filler powder is Al_2O_3. The activator powder used is a halogen compound, such as AlF_3, forming approximately 3% by weight. The activator powder is therefore a halide compound of the donor metal Al.

The mean particle size of the inert filler powder is approximately equal to the mean particle size of the metal donor powder, being 150 μm. The proportion of the donor metal Al in the metal donor powder, which is an alloy, is 50% by weight. The specific density of a powder pack mixture is high not because of a high metal donor powder content, but rather because of the selected particle size distribution. With this bed of the powder pack mixture, there is sufficient permeation by the coating gases emanating from the halide compound.

To coat the inner surface of the turbine guide vane, the powder mixture which has been prepared in this manner is introduced into its cavity. At the selected particle size distribution of the inert filler powder and of the metal donor powder, the bed has sufficient flow properties and gains

access even to the tight corners of the cavity. The subsequent coating is performed at 1080°C for a holding time of 6 h. It may be performed at the same time as the external coating, i.e., the coating of the outer surface of the turbine guide vane, which may be performed using a conventional powder pack process or also using a gas diffusion coating process. Generally, the coating is performed on a plurality of turbine guide vanes simultaneously.

The Al content in the internal coating which has been deposited in this manner is between 30 and 35% by weight and consequently in a highly advantageous range, i.e., there is, for example, no embrittlement of the layer.

Even in tight corners or recessed regions of the cavities, the layer thicknesses are in the range of 60 to 110 μm, thus ensuring that the internal coating function has protection against oxidation and corrosion.

ABSTRACT

A process for coating hollow bodies, in which a powder mixture including a metal donor powder, an inert filler powder and an activator powder including a metal halide is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the hollow body and is heated, in which process, in order to increase the internal layer thicknesses, the inert filler powder is provided with a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

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PROCESS FOR COATING HOLLOW BODIES

The invention relates to a process for coating hollow bodies, in which a powder mixture comprising a metal donor powder, an inert filler powder and an activator powder is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the body, e.g. comprising an Ni-, Co- or Fe-base alloy, and is heated.

The known processes for the diffusion coating of components made from heat-resistant alloys, such as Ni-, Co- or Fe-base alloys, include what are known as the powder pack processes. A process of this type is disclosed in US 3,667,985, in which the component surfaces to be coated are brought into contact with a donor powder comprising titanium and aluminum, to which an inert filler material and a halide activator are admixed, and is heated. US 3,958,047 has disclosed a powder pack process in which the metallic component is brought into contact with a donor powder which contains aluminum and chromium and is diffusion-coated by heating.

These processes are particularly suitable for coating the outer surfaces of metallic components, producing layer thicknesses of between 50 and 100 μm. However, the processes have inherent drawbacks when coating internal surfaces, and consequently the internal layer thicknesses which are achievable with relatively complicated geometrical forms with narrow gaps, tight angles or undercuts are limited and inadequate, generally being below 30 μm. A problem in this respect is that the donor powders have only a low ability to flow and therefore do not sufficiently fill the cavities. Moreover, after the coating, the donor powder can only be removed from the cavities with difficulty, and it is not possible to avoid leaving residues, and also the donor powder sinters to the surfaces.

The abovementioned drawbacks of the powder pack processes can in part be eliminated using what are known as gas diffusion coating processes. One process of this type is known from US 4,148,275, in which a powder mixture which contains, for example, aluminum is arranged in a first chamber and the metallic components to be coated are arranged in a second chamber of a vessel. The coating gas is generated by heating the powder and, using a carrier gas, is deposited on the outer and inner surfaces of the components to be coated. However, the gas diffusion coating processes have the drawback that the devices for carrying out the process, such as for example for the forced guidance of the coating gases, are complex and expensive compared to those used for the powder pack processes. Furthermore, in this case too, the internal layer thicknesses which can be achieved are limited, since the coating gas or the donor metal gas is depleted on its route through the cavities of the component and a layer thickness gradient is produced along the length of the cavity. Since process conditions mean that the layer thickness of the outer coating is greater than that of the inner coating, the service life of the component is limited on account of the thinner internal coating.

US 4,208,453 has disclosed a process for the diffusion coating of the inner and outer surfaces of components, such as gas turbine blades, in which a powder mixture comprises 10% of chromium donor powder with a particle size of from 10 to 20 μm and 90% of alumina granules with a particle size of from 100 to 300 μm. In addition, a metal halide is added as activator. This disclosure does not deal with measures for increasing the layer thickness in cavities of complicated geometry.

DE 30 33 074 A1 discloses a process for the diffusion coating of the inner surface of cavities, in which a metallic workpiece can be coated with an aluminizing diffusion powder mixture comprising 15% of aluminum powder with a particle size

of 40 μm and 85% of alumina powder with a particle size of approximately 200 to 300 μm, as well as an NH_4Cl powder.

US 5,208,071 discloses a process for aluminizing a ferritic component with an alumina slurry, followed by heat treatment, the slurry comprising at least 10% by weight of chromium, at least 10% by weight of inert filler material, at least 12% by weight of water, a binder and a halogen activator, and finally the coated ferritic component is heat-treated. The process technology involved means that the use of a slurry differs significantly from a powder pack coating process.

GB 2 109 822 A has disclosed a metal diffusion process with which diffusion coatings can be produced more quickly than in the powder pack process, the coating powder being in loose form and being kept in contact with the component to be coated, in particular including with its internal surface, by mechanical means during the heating. The composition of the coating powder may comprise 10 to 60% of chromium powder, 0.1 to 20% of chromium halide and alumina.

The problem on which the present invention is based is that of improving a powder pack process of the generic type described in the introduction in such a way that the layer thicknesses of the internal coating are sufficiently great even in the case of cavities with relatively complex geometries.

According to the invention, the solution to this problem is characterized in that the inert filler powder is provided with a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

The advantage is that, when the particle sizes are selected in this way, it is possible to increase the specific density without agglomeration of the powder mixture, for example on account of an excessively high metal donor powder content. It is also ensured that there is no premature depletion of the

donor metal. A powder mixture of this type has good flow properties and, in tight corners, gains access to internal cavities which are to be coated. It is possible to coat hollow bodies, such as guide vanes and rotor blades of gas turbines made from heat-resistant Ni-, Co- or Fe-base alloys. Even in tight corners or recessed regions of the cavities, the layer thicknesses of the internal coating lie in the range from 50 to 110 μm, therefore ensuring that the internal coating functions as an oxidation-resistant and corrosion-resistant layer.

In a preferred configuration, the metal donor powder and the inert filler powder are provided with a mean particle size of greater than 40 μm, so that it is possible to achieve good permeation of the coating gas through the bed of the powder mixture.

The powder mixture is preferably provided with a metal donor powder content of 10 to 25% by weight, in order to prevent agglomeration of the powder mixture and to ensure good permeation through the bed.

Furthermore, it is expedient for an alloy with a donor metal content of 20 to 80% by weight to be provided as the metal donor powder, so that a sufficiently great layer thickness is ensured on account of the high donor metal content.

It may be advantageous for a mixture of an alloy with a donor metal content of 40 to 70% by weight and an alloy with a donor metal content of 30 to 50% by weight to be provided as the metal donor powder, so that the depletion of the metal donor in the two alloys takes place in steps, i.e. with a time delay.

The metal donor powder and the inert filler powder may be provided with a mean or average particle size of 150 μm. A powder mixture of this type has good flow properties and fills

The cavity has a length of approximately 160 mm. Its inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite end sections. To coat the inner surfaces of the guide vanes, a powder mixture comprising approximately 20% by weight of metal donor powder and approximately 80% by weight of inert filler powder is provided. AlCr is selected as the metal donor powder, and Al_2O_3 is selected as the inert filler powder. The melting point of AlCr is at least approximately 100°C higher than the coating temperature of approximately 800°C - 1200°C, so that there is no diffusion bonding of the metal particles to one another or agglomeration.

An activator powder forms approximately 3% by weight, the powder selected being AlF_3, i.e. a halide compound. Another example of a suitable activator powder is $CrCl_3$. A compound of this type has to have a low vapour pressure at the coating temperature, so that it is retained throughout the entire coating process. Moreover, a halide compound of the donor metal, in this case aluminum, is used, in order to avoid agglomeration as a result of a chemical reaction of the halogen with the donor metal.

The aluminum content, i.e. the metal donor content, in the metal donor powder is 50% by weight.

The powder mixture which has been prepared in this way is introduced into the cavity of the guide vanes for the purpose of coating the internal surfaces. The subsequent coating takes place at 1080°C with a holding time of 6 h, while the external coating, i.e. the coating of the outer surfaces of the guide vane, can take place simultaneously in a single-stage process using a conventional powder pack process or alternatively by means of a gas-diffusion coating process.

In the internal coating which is deposited in this way, the Al content in the layer is between 30 and 35% by weight.

The mean particle size of the inert filler powder is 100 μm, and is significantly greater than the particle size of the metal donor powder, which is 60 μm. The aluminum content, i.e. the metal donor content, in the metal donor powder is 50% by weight.

The powder mixture which has been prepared in this way is introduced into the cavity of the guide vanes for the purpose of coating the internal surfaces. The subsequent coating takes place at 1080°C with a holding time of 6 h, while the external coating, i.e. the coating of the outer surfaces of the guide vane, can take place simultaneously in a single-stage process using a conventional powder pack process or alternatively by means of a gas diffusion coating process.

In the internal coating which is deposited in this way, the Al content in the layer is between 30 and 35% by weight.

In a second example, an inert filler powder (Al_2O_3) with a mean particle size of approximately 100 μm is once again selected, forming approximately 80% by weight of the powder mixture. As activator powder, AlF_3 forming approximately 3% by weight of a powder mixture is selected and admixed.

Unlike in Example 1, the metal donor powder, which forms approximately 20% by weight of the powder mixture, comprises two fractions. The first fraction is an alloy comprising AlCr, in which the aluminum content is 50% by weight. In the second fraction, the donor metal content, i.e. the aluminum content, is lower, being 30% by weight. This measure can be used to optimize the coating process in such a manner that first of all the fraction with the lower Al content is depleted, but the coating process is continued by the fraction with the higher Al content. In this way, it is possible to increase the ductility of the layers on the inner surfaces of the guide vane.

The Al content in the inner layers is 24 to 28% by weight. The inner layer thicknesses are between 65 and 105 μm and are therefore significantly above the layer thicknesses which can be achieved with the conventional (powder pack) processes.

In a third example, the hollow body is a hollow turbine guide vane of a gas turbine, which is provided with an oxidation-resistant and corrosion-resistant layer by means of a powder pack coating process. The elongate cavity is approximately 180 mm long. The inner surfaces are spaced apart at between 2 and 6 mm and converge at two opposite, longitudinal-side end sections. To coat the inner surface of the guide vane, a powder mixture comprising approximately 15% by weight of metal donor powder and just below 85% by weight of inert filler powder is provided. Depending on the particular application, the metal donor powder content may lie in the range from 10 to 25% by weight. The metal donor powder is AlCr, and the inert filler powder is Al_2O_3. The activator powder used is a halogen compound, such as AlF_3, forming approximately 3% by weight. The activator powder is therefore a halide compound of the donor metal Al.

The mean particle size of the inert filler powder is approximately equal to the mean particle size of the metal donor powder, being 150 μm. The proportion of the donor metal Al in the metal donor powder, which is an alloy, is 50% by weight. The specific density of a powder pack mixture is high not because of a high metal donor powder content, but rather because of the selected particle size distribution. With this bed of the powder pack mixture, there is sufficient permeation by the coating gases emanating from the halide compound.

To coat the inner surface of the turbine guide vane, the powder mixture which has been prepared in this way is introduced into its cavity. At the selected particle size distribution of the inert filler powder and of the metal donor powder, the bed has good flow properties and gains access even

to the tight corners of the cavity. The subsequent coating takes place at 1080°C for a holding time of 6 h. It may be carried out at the same time as the external coating, i.e. the coating of the outer surface of the turbine guide vane, which may take place using a conventional powder pack process or also using a gas diffusion coating process. Generally, the coating is carried out on a plurality of turbine guide vanes simultaneously.

The Al content in the internal coating which has been deposited in this way is between 30 and 35% by weight and consequently in a highly advantageous range, i.e. there is, for example, no embrittlement of the layer.

Even in tight corners or recessed regions of the cavities, the layer thicknesses are in the range from 60 to 110 μm, thus ensuring that the internal coating function has protection against oxidation and corrosion.

Patent claims

1. A process for coating hollow bodies, in which a powder mixture comprising a metal donor powder, an inert filler powder and an activator powder comprising a metal halide is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the hollow body and is heated, characterized in that the inert filler powder is provided with a mean particle size which is approximately the same as the mean particle size of the metal donor powder in that the metal donor powder and the inert filler powder are provided with a mean particle size of greater than 40 μm, and in that a powder mixture with a metal donor powder content of 10 to 25% by weight is provided.

2. The process as claimed in claim 1, characterized in that an alloy with a donor metal content of 20 to 80% by weight is provided as the metal donor powder.

3. The process as claimed in claim 1 or 2, characterized in that a mixture of an alloy with a donor metal content of 40 to 70% by weight and an alloy with a donor metal content of 30 to 50% by weight is provided as the metal donor powder.

4. The process as claimed in one or more of the preceding claims, characterized in that a powder mixture with an activator powder content of 2 to 5% by weight is provided.

5. The process as claimed in one or more of the preceding claims, characterized in that a metal halide of the donor metal is selected for the activator powder.

6. The process as claimed in one or more of the preceding claims, characterized in that AlCr is selected as the donor metal powder.

7. The process as claimed in one or more of the preceding claims, characterized in that the metal donor powder and the inert filler powder are provided with a mean particle size of approximately 150 μm.

Abstract

A process for coating hollow bodies, in which a powder mixture comprising a metal donor powder, an inert filler powder and an activator powder comprising a metal halide is provided, the powder mixture is brought into contact with an inner surface, which is to be coated, of the hollow body and is heated, in which process, in order to increase the internal layer thicknesses, the inert filler powder is provided with a mean particle size which is approximately the same as the mean particle size of the metal donor powder.

DECLARATION AND POWER OF ATTORNEY

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am an original, first and joint inventor of the subject matter that is claimed and for which a patent is sought on the invention entitled **PROCESS FOR COATING HOLLOW BODIES**, the specification of which was filed as International Application No. PCT/DE99/03942, on December 9, 1999, an English translation of which is enclosed herewith.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims.

I acknowledge the duty to disclose information that is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate filed by me on the same subject matter having a filing date before that of the application on which priority is claimed:

PRIOR FOREIGN APPLICATION(S)

(Number)	(Country)	(Day/month/year filed)	Priority Claimed Under 35 USC 119	
198 56 901.7	Fed. Rep. of Germany	10 December 1998	Yes X	No _

And I hereby appoint Richard L. Mayer (Registration No. 22,490) my attorney with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

EV003629170

Please address ~~all~~ communications regarding this application to:

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PATENT TRADEMARK OFFICE

I hereby declare that all statements ~~made herein~~ of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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